

Filament Lifetime and Chemical Performance

Introduction

Filaments are important consumables for gas chromatograph-mass spectrometry (GC/MS) analyses. Filaments, which tend to get overlooked, are key to generating ions in the source and can affect data quality, reproducibility, and accuracy. Choosing the best filament for the instrument source is important for maintaining the longest lifetime and best chemical performance for GC/MS. This technical overview summarizes the results of lifetime and chemical testing of filaments from different manufacturers.

Experimental

Electron ionization (EI) filaments for the Agilent 5977B GC/MSD were procured from Agilent (part number G7005-60061) and another vendor, noted as "vendor A". An Agilent 8890 GC system was coupled with an Agilent 5977B MSD with a 30 m Agilent J&W DB-5ms Ultra Inert column (part number 122-5532UI) and was used for lifetime testing. The GC parameters were held at constant flow and temperature during lifetime testing, as no injection was made. The lifetime testing only focused on the filaments and the MS source.

For chemical performance testing, the Agilent Intuvo 9000 GC was configured with an Agilent split/splitless inlet liner and Guard Chip, a single MS flow path, and a 30 m DB-5ms Ultra Inert column. The 5977B GC/MSD was installed with a 3 mm drawout plate. Octafluoronaphthalene (OFN) at 100 fg/ μ L was procured from Agilent (part number 5188-5347) for chemical performance testing.

Lifetime testing

An extractor tune (etune.u) was run when the GC/MS system was confirmed leak-free with water and oxygen ratios to m/z 69 under 2%. Any high presence indicates a leak, which will greatly shorten filament life and stability. A macro was designed to cycle the filament on and off, and monitor helium (m/z 4), water (m/z 18), and oxygen (m/z 32) every 100 cycles. The starting helium abundance was noted before beginning the test for each filament; then, the macro was run for 24 hours, and helium abundance was reviewed. The helium abundance was tracked until the filament failed or the abundance was <10% of the original value, when the filament was considered spent.

Chemical performance testing

Table 1 summarizes GC method settings, consumables, and MS conditions for chemical performance testing. A sequence of 228 injections of OFN was run to review filament effect on chemical performance. Twelve individual runs of OFN were used to generate instrument detection limit (IDL) values, resulting in 19 data sets per tested filament.

Results and discussion

Table 2 summarizes the average filament lifetime from the life cycle testing. Agilent filaments had an average lifetime of 94 hours in this extreme testing with a narrow RSD, while the other tested filament only lasted 16 hours on average with a wider RSD. Agilent filaments lasted almost six times longer than the other tested filaments, on average.

Table 1. GC and MSD instrument conditions and consumables.

Parameter	Value
Injection Volume	1 μ L
Inlet	Split/splitless 250 °C Pulsed splitless 25 psi until 0.5 min Purge 100 mL/min at 1.0 min Switched septum purge 3 mL/min
Inlet Liner	Agilent inlet liner, Ultra Inert, splitless, double taper (p/n 5190-3983)
Guard Chip and Temperature	Agilent Guard Chip, Intuvo, split/splitless inlet (p/n G4587-60565) Track oven temperature
Bus Temperature	200 °C
Column	Agilent J&W DB-5ms Ultra Inert, 30 m \times 0.25 mm, 0.25 μ m (p/n 122-5532UI-INT)
Column Temperature Program	45 °C (hold for 2.25 min), 40 °C/min to 200 °C (hold for 2 min)
Carrier Gas and Flow Rate	Helium at 1.20 mL/min, constant flow
Intuvo Flow Path Connections	Agilent Flow Chip, Intuvo, inlet (p/n G4581-60031) Agilent Flow Chip, Intuvo, D2-MS (p/n G4581-60033) Agilent detector tail, Intuvo, MS (p/n G4590-60009)
Transfer Line Temperature	250 °C
Ion Source Temperature	250 °C
Quadrupole Temperature	150 °C
MS Acquisition Mode	Selected ion monitoring (SIM)
SIM Ions (Dwell Time)	m/z 272 (dwell 25 ms)

Table 2. Average lifetime (hours) and %RSD for tested filaments.

Filament Manufacturer	Average Lifetime (Hours)	%RSD
Agilent	94.2	9.0 %
Vendor A	16.0	35.0 %

For chemical performance testing with OFN, 10 fg is the maximum allowable instrument detection limit (IDL) and values at 10.0 fg or lower are considered a "pass" for the filament. Each filament had 19 data sets and all IDL values are mapped out in Figure 1. Ideally, a well-performing filament will pass the IDL check for all 19 data sets (228 total runs). An aging filament may show an increase in IDL value with more runs, while an inconsistent filament will have varied IDL values below and above 10 fg. Looking at Figure 1, the IDL values for Agilent filaments (blue dots) all fall under 10 fg, indicating consistent performance across the tested filaments. Comparatively, the IDL values for vendor A (orange dots)

include nine IDL values at or above the 10 fg limit, indicating varied performance across the vendor A filaments. Capability analysis of both Agilent and vendor A filaments was completed with an upper boundary limit of 10.0. For vendor A, 14% of the data falls outside of the upper limit with a sample mean of 7.1 fg and standard deviation of 2.54 fg, while Agilent filament data are below the 10 fg upper limit with a sample mean of 5.4 fg and standard deviation of 1.8 fg. These results indicate more consistent performance for Agilent filaments. Consistent filament performance is important for consistent compound fragmentation, ion ratios, and overall data quality and accuracy.

Conclusion

Agilent filaments displayed consistent performance for lifetime and IDL. All data sets for the Agilent filaments performed under the upper IDL limit of 10 fg, while 14% of the data from vendor A filaments tested above the upper IDL limit. Overall, Agilent filaments lasted approximately six times longer than vendor A filaments, and are the optimal choice for MS filament needs.

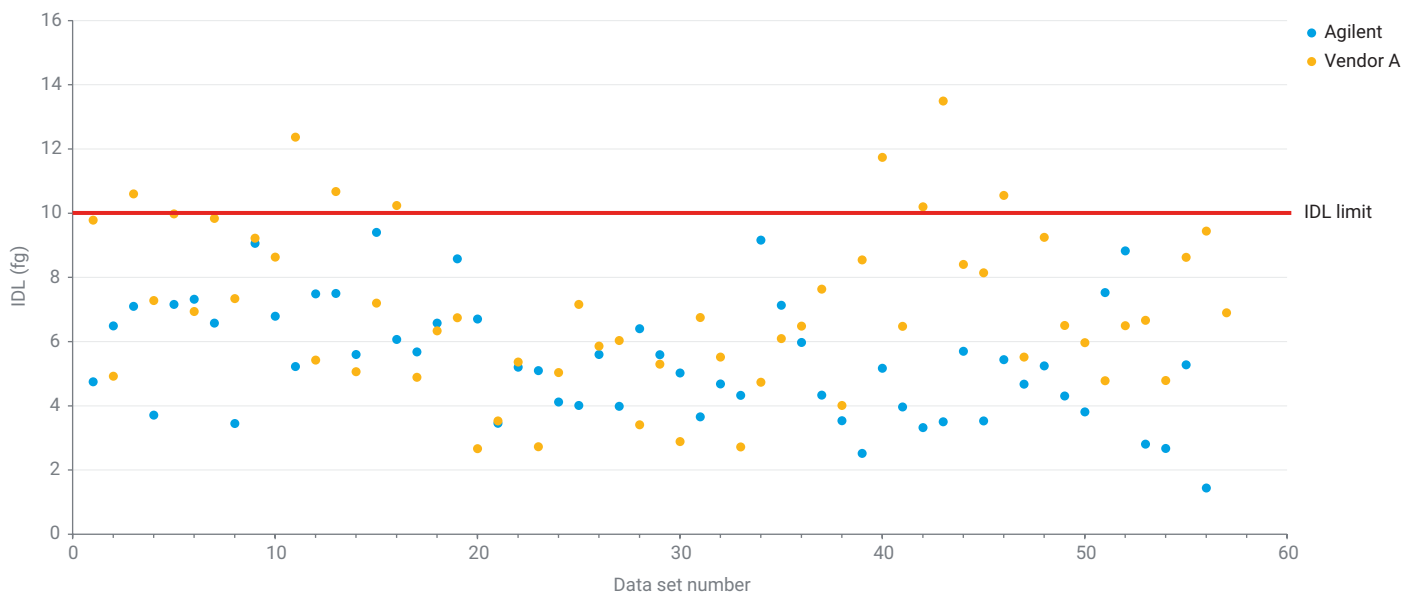


Figure 1. OFN IDL values for Agilent filament data sets (blue dots) and Vendor A filament data sets (orange dots) with the upper limit of 10 fg, indicated with the red line.

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